PATENT SPECIFICATION

736.303



Date of Application and filing Complete Specification April 8, 1952. No. 8998/52.

Application made in United States of America on May 17, 1951.

Application made in United States of America on Sept. 20, 1951.

Complete Specification Published Sept. 7, 1955.

Index at acceptance:—Class 2(2), A4C(1:2:3).

COMPLETE SPECIFICATION

Improvements in or relating to the Treatment of Cellulose Ethers and the Resulting Products

We, Hercules Powder Company, a corporation organized under the laws of the State of Delaware, one of the United States of America, of 900 Market Street, City of Wilmington, State of Delaware, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the treatment of cellulose ethers and to the resulting products.

In a more specific aspect, this invention relates to a process for the removal of color 15 bedies from a cellulose ether by a novel process and to the resulting colorless cellulose ether.

Thermoplastic compositions based on ethyl cellulose and similar thermoplastic ethers of 20 cellulose have been found to be useful where the properties of toughness, dismensional stability and flexibility are required. However, when such cellulose ethers have been molded into useful objects at high temperatures, a considerable discoloration of the cellulose ether composition takes place. Thus, compositions depositing a substantially colorless mass from solution have given relatively dark amber masses much like tortoise shell upon being 30 molded under heat. In addition, there has been considerable loss of viscosity; i.e., degradation, leading to brittle molded products.

Some attempts have been made to improve color by treatment with various bleaching 35 agents and by treatment with acids. Where such improvements in color have been retained in the molding operation, they have resulted in such extreme loss of viscosity that a relatively useless product results. As a result, it 40 has not been possible to prepare clear colorless molded products of cellulose ethers or even to prepare molded cellulose ether articles in pale or pastel shades. Molded cellulose ether articles have therefore been made 45 primarily in pigmented or dark colors.

In our copending Application No. 9459/52, (Serial No. 736,304) we disclose a method of bleaching and removing color bodies from an

[Price 3s. Od.]

organic solvent-insoluble cellulose ether which comprises contacting a slurry of said cellulose 50 ether in an organic solventinwhichthe cellulose ether does not dissolve, said solvent being inert to both inorganic water-soluble chlorites and hypochlorites under the conditions used in the process, miscible with water and capable of 55 swelling said cellulose ether, with a water-soluble inorganic chlorite, when the ph of said slurry is lower than 6, or with a water-soluble inorganic chlorite activated with a water-soluble inorganic hypochlorite, when the ph 60 of said slurry is at least 6, and recovering the bleached cellulose ether from the accompanying liquid.

In our British Specification No. 634,808, we disclose a process for stabilizing cellulose 65 ethers by adding to or incorporating with the cellulose ether, diisobutylphenol. According to this process, the cellulose ether may be suspended in a swelling medium such as aqueous alcohol and a solution of the stabilizer added 70 to the suspension for absorption by the cellulose ether. A similar process is disclosed in our British Specification No. 643,348 wherein the stabilizer is paracyclohexylphenol and sulphur dioxide or a compound releasing sulphur-75 dioxide upon application of heat.

British Specification No. 580,359 discloses the stabilization of a cellulose ether with a polyhydroxy benzene. The ether may be mixed for 1 or 2 hours with a solution of 80 stabilizing agent in an organic liquid which has a swelling action on the ether, after which the liquid is removed. A slightly acid treatment medium may be used for the precipitation and washing of the ether containing the 85 phenoli.

A process has also been proposed for improving the color and color stability of cellulose ethers which comprises subjecting the cellulose ether in particulate form to extract of swelling the cellulose ether but is incapable of swelling the cellulose ether but is incapable of dissolving more than a small proportion thereof, and then incorporating with the cellulose ether a color stabilizer comprising citric 95 acid or an organic sulphite,

novel process for producing colorless cellulose ethers of low alkaline content and having improved viscosity and color stabilities.

In accordance with this invention, it has been found that novel color-stable and viscosity-stable cellulose ethers can be prepared by contacting an organic solvent-soluble cellulose ether with an acidified aqueous swelling 10 medium, separating said cellulose ether from said swelling medium, stabilizing the cellulose ether by adding an unacidified aqueous swelling medium and a minor proportion of a phenol and separating said cellulose ether from 15 said unacidified swelling medium, the phenol being absorbed in the cellulose ether.

The cellulose ethers that are employed in practising this invention are organic solventsoluble, i.e., they are soluble in any of the 20 common organic solvents such as acetone, benzene, toluene-alcohol, methanol, ethanol, ethyl acetate, butyl acetate, and the like. The primary factor that determines whether or not a cellulose ether is organic solvent-soluble is the 25 degree of substitution of the cellulose ether, and the extent of etherification necessary to produce an organic solvent-soluble cellulose ether is known to those skilled in the art. For example (ethyl cellulose having an ethoxyl con-30 tent between about 37% and about 52%, pre-ferably between about 43% and about 48%, and having a viscosity of at least 20 cps., is particularly suitable. One method of preparing such an ethyl cellulose involves the pre-35 paration of an alkali cellulose by contacting a cellulosic material with strong caustic. The resulting alkali cellulose is treated in an autoclave under pressure with an excess of ethyl chloride and the resulting mixture is then dis-40 tilled to remove ether and alcohol by-products as well as unreacted ethyl chloride. The resulting ethyl cellulose is then water-washed. In addition to ethyl cellulose, other cellulose ethers can be treated in accordance with this 45 invention. For example, the process is applicable to the bleaching of propyl cellulose, ethyl propyl cellulose, ethyl butyl cellulose, methyl ethyl cellulose, benzyl cellulose, and the like. In addition to the alkyl and aralkyl cellulose

50 ethers described above, the invention is generally applicable to organic solvent-soluble cellulose ethers. For example, ethyl hydroxyethyl cellulose can be treated in accordance with the invention to produce a color-free, color-stable 55 product. Also ether-ester derivatives of cellulose can be treated in accordance with this process, but in these ether-ester derivatives the ether grouping is the dominant substituent and the ester grouping is present only in a minor 60 amount. However, the degree of substitution of the ether-ester derivative is such that the derivative is soluble in the organic solvents named above. Throughout the specification and claims the term "cellulose ether" will be 85 employed in the description of the invention.

It is an object of this invention to provide a It will be understood that this term includes those ether-ester cellulose derivatives wherein the ether substituent is the dominant substituent and the ester substituent is present only in a minor amount as well as unesterified 70 cellulose ethers.

Various aqueous media can be used to effect the desired swelling of the cellulose ether. For example, the swelling of the cellulose ether can be effected by aqueous digestion of the ether 75 at an elevated temperature and pressure. Long steeping of the cellulose ether in water at 0 to 3° C. also will produce swelling. Preferably the swelling is effected in the presence of an aqueous solution of an organic solvent 80 for the ether, the concentration of the solution being such that the cellulose ether is substantially insoluble in the solution. Organic solvents that can be used are acetone, dioxane, tetrahydrofuran, methyl acetate, ethyl acetate, and the like. The preferred organic solvents are the lower water miscible aliphatic alcohols, for example, methanol, ethanol, propanol, isopropanol, and the like, with isopropanol being the most preferred organic solvent. In general, 90 the aqueous solution of the organic solvent will contain from 10% to 50% by weight of the When isopropanol is the organic solvent. organic solvent, a concentration of 30% to 35% by weight is preferred, and when ethanol 95 is used, the preferred concentration is within the range of 40% to 50% by weight. The treatment of the cellulose ether with the aqueous swelling medium results in a penetration of the ether granules by the swelling 100 medium and in a deashing of the ether to remove inorganic constituents, such as alkali metal and alkaline earth metal compounds (e.g. their hydroxides and salts) and alkali metals and alkaline earth metals chemically bound to 105 functional groups in the cellulose ether and such impurities as iron, copper and low molecular weight organic materials. For effective deashing, a swelling medium which has been acidified until the pH is substantially below 6 110 and preferably within the range of 1.5 to 3.5 is employed. A pH higher than 3.5 can be used, but poor deashing of the cellulose ether results, and at a pH lower than 1.5 the cellulose ether has a tendency to degrade.

In general, the deashing of the cellulose ether is carried out at room temperature, but, if desired, elevated temperatures of 75° C. and higher can be used. The contact time for the deashing step is variable, but a contact time 120 within the range of 0.25 to 5.0 hours is preferred to effect the desired deashing of the cellulose ether. However, a contact time up to 20 hours and longer can be used, if desired. In Example 2 a contact time of 16 hours was 125 employed.

Either simultaneously with or subsequent to the deashing and swelling procedure the cellulose ether may be treated with a chlorite 130 bleaching agent.

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The following examples describe the preferred conditions for carrying out the process when using a chlorite bleaching agent, but operating conditions outside the preferred 5 ranges can be used, if desired. For example, temperatures within the range of 0° to 130° C. and contact times within the range of 0.1 to 10 hours are within the scope of the invention.

The bleaching agent that is employed is a 10 water-soluble inorganic chlorite, preferably a metal chlorite, but non-metallic chlorites, such as ammonium chlorite, can be used. The preferred metal chlorites are the alkali metal and alkaline earth metal chlorites, for example, 15 potassium chlorite, calcium chlorite, magnesium chlorite and most preferably sodium chlorite. The chlorite bleaching agent can be added to the cellulose ether-swelling medium For example, it can be in various forms. 20 added either in the solid form or in the form of an aqueous solution containing from 1 to 25% by weight of the chlorite. The amount of chlorite that is employed is within the range of 0.1 to 2% by weight based upon the cellu-25 lose ether, but in most instances quantities in excess of 0.2% by weight are not necessary.

Various acidic materials can be employed to effect the desired pH adjustment in the deashing operation. For this pH adjustment the 30 inorganic acids are preferred, and the most preferred acids are the strong mineral acids such as hydrochloric acid, sulfuric acid, and

nitric acid.

After the desired deashing of the cellulose 35 ether has been effected, the cellulose ether is separated and recovered from the resulting mixture. Suitable methods of recovering the cellulose ether are centrifuging, filtration, decanation or draining the solid cellulose ether 40 from the accompanying liquid. The recovered cellulose ether is then washed with either water or additional amounts of aqueous swelling medium. The latter is preferred for the washing operation, and a series of washing steps is 45 usually necessary in order to effect the removal of salts, excess acid and undesirable low molecular weight degraded cellulose material. The washing is continued and it is considered complete when the pH of the effluent liquor from 50 the washing step has a pH within the range of 4.2 to 4.8. Further washing of the cellulose ether may be carried out, but in most instances such further washing is not necessary. The resulting cellulose ether is substantially free of 55 alkaline constituents and has an ash content not in excess of 0.02% by weight.

The cellulose ether resulting from the deashing operation is subject to rapid oxidation and depolymerization upon exposure to heat 60 unless properly stabilized. Therefore, during the washing of the bleached cellulose ether with aqueous swelling medium, a phenolic stabilizer is added to the cellulose ether in the Various phenols are suitable for last wash. 65 effecting the desired stabilization of the de-

ashed cellulose ether. Among these stabilizers are p-sec-amylphenol, di-sec-amylphenol, osec-amylphenol, p-cyclohexylphenol, diisobutylphenol, p-tert-amylphenol, methylphenol, tertbutyl-p-cresol, methylene bis(tert-butyl-p- 70 cresol), methylene bis-p-cresol, di(hydroxy methyl phenyl) methyl-p-cresol, and the like. In the specific examples di-tert-amylphenol was employed as a stabilizer. The amount of phenolic stabilizer employed to effect the 75 desired stabilization is within the range of 0.5% to 1% by weight based on the cellulose ether. As indicated above, the phenolic stabilizer can be dissolved in the swelling medium that is employed as the last washing medium for the 80 deashed cellulose ether, or alternatively a solution or dispersion of the phenolic stabilizer can be added to the mixture while the deashed cellulose ether is undergoing its last wash with swelling medium.

After deashing and stabilizing the cellulose ether, the resulting cellulose ether is recovered by draining off the last wash liquid and drying the recovered cellulose ether. This cellulose ether can be processed into plastics pro- 90 ducts lighter in color than those possible heretofore. Also, a wide range of dyes can be used with this cellulose ether, and the resulting products are essentially free from odor. Prior to using the cellulose ether resulting from this 95 process in a molding operation, it is preferred to add additional quantities of phenolic stabilizer in order to prevent oxidation or depolymerization of the ether. Preferably the resulting product contains from 1% to 3% by 100 weight of phenolic stabilizer based upon the cellulose ether.

Further improvement in the color stability may be obtained by contacting the cellulose ether with a water-soluble inorganic chlorite 105 bleaching agent in the presence of the acidified aqueous swelling medium.

The following examples are illustrative of this invention.

EXAMPLE 1. To 5000 ml. of aqueous 35% by weight isopropanol was added 1:1 hydrochloric acid in an amount sufficient to produce a pH of 2.2. The dilute isopropanol was prepared from dis- 115 tilled isopropanol and distilled water. acidified aqueous alcohol was added 681 grams of ethyl cellulose. The ethyl cellulose contained 46.5% ethoxyl groups and when dissolved in a solvent consisting of 80 parts 120 toluene and 20 parts ethanol, to give a 5% solution, the solution possessed a viscosity of 97 cps. at 25° C. The mixture of ethyl cellulose, swelling medium and acid was heated to 75° C. and maintained at this temperature for 125 1.5 hours, and then the liquor was drained off. The volume was restored by the addition of 4000 ml. of fresh neutral 35% isopropanol. After 0.5 hour at room temperature the liquor was drained off again. The volume was 130

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restored with 4000 ml. of fresh 35% neutral isopropanol. This procedure was repeated three more times to wash the ethyl cellulose free of salts and the excess acid. Then to the last wash was added 5 grams of di-tert-amylphenol dissolved in 25 ml. of 35% isopropanol. The liquor was drained off and the residual material was vacuum dried.

The ethyl cellulose then was tested for powder with the use of a two-roll mill with roll temperatures of 280° F. to 310° F. and rolling time of 20 minutes. The formulation consisted of ethyl cellulose, a methyl styrene 15. plasticizer, and dissobutyl phenol in the ratio of 85:15:2. The colloided plastic was cut into pellets, dried for 2 hours at 100° C., and then injection molded at 410° F. into plastics disks with the use of a 1-ounce injection mold-20 ing machine. The injection molded disks were 24 inches in diameter and 0.180 inch in thickness. The color or chromaticity and clarity or haze of the disks were measured with a photo-electric filter photometer. Apparatus 25 suitable for measuring color or chromaticity is described by R. H. Osborn in U.S. Patent 2,382,439, and a similar device modified to permit the clarity measurements was used to measure the color and clarity of the treated measure the color and clarity of the treated measurement and untreated ethyl cellulose. The color 30 and untreated ethyl cellulose. measurements are expressed in Cellulose Products Standard Color Scale (CSCS) units and the clarity is expressed in per cent. haze. The color and clarity of air are zero with this 35 measuring device, and the ethyl cellulose samples having the lowest color and per cent. haze are the most desirable. The following data were observed:-

% Haze Color Ethyl cellulose product -4.3 5.1 40 Untreated ethyl cellulose -

Example 2

To 5000 ml. of aqueous 35% by weight iso-propanol was added 5 ml. 1:1 hydrochloric 45 acid. The dilute isopropanol was prepared from distilled isopropanol and distilled water. To the acidified aqueous alcohol was added 681 grams ethyl cellulose: The pr of the mixture was 3.0. The ethyl cellulose contained 50 47.0% ethoxyl groups, and when dissolved in a solvent consisting of 80 parts toluene and 20 parts ethanol, to give a 5% solution, the solutien possessed a viscosity of 41 cps. at 25° C. The mixture of ethyl cellulose, swelling 55 medium and acid was allowed to stand for 16 hours at room temperature. Then the liquor was drained off. The volume was restored by the addition of 4000 ml. fresh neutral 35% isopropanol. After 0.5 hour at 60 recm temperature the liquor was drained off again. This procedure was repeated two more times to wash the ethyl cellulose free of excess salts and the excess acid. To the last wash was added 5 grams di-tert-amylphenol dis-

solved in 200 ml. 35% isopropanol. After one 65 hour the liquor from the last wash was drained off and the material was vacuum dried. The following data were observed in examining the treated and untreated ethyl cellulose as 70 described in Example 1:-

Color % Haze Ethyl cellulose product -6.8 19 Untreated ethyl cellulose - 14 22

Example 3.

To 5000 ml of aqueous 35% by weight iso-75 propanol was added 12 ml. 1:I hydrochloric The dilute isopropanol was prepared from distilled isopropanol and distilled water. To the acidified aqueous alcohol was added 681 grams of ethyl cellulose. The pr of the 80 mixture was 2.2. The ethyl cellulose contained 45.5% ethoxyl groups, and when dissolved in a solvent consisting of 80 parts toluene and 20 parts ethanol, to give a 5% solution, the solution possessed a viscosity of 94 cps. at 25° C. 85 The mixture of ethyl cellulose, swelling medium and acid was heated to 75° C. The mixture was maintained at 75° C. for 1.5 hours. Then the liquor was drained off. The volume was restored by the addition of 4000 90 ml. of 35% isopropanol. After 0.5 hour at room temperature the liquor was drained off again. The volume was restored with 3000 ml. fresh neutral 35% isopropanol. This procedure was repeated seven more times to wash the 95 ethyl cellulose free of the salts and the excess acid. Then to the last wash was added 5 grams di-tert-amylphenol dissolved in 25 ml. 35% isopropanol. The liquor was drained off and the material was vacuum dried. The follow-100 ing data were observed in examining the treated and untreated ethyl cellulos as described in Example 1:—

% Haze Color 105 Ethyl cellulose product -5.5 5.3 Untreated ethyl cellulose - 17

EXAMPLE 4.

To 5000 ml. of aqueous 35% by weight isopropanol was added 12 ml. 1:1 hydrochloric acid. The dilute isopropanol was prepared 110 from distilled isopropanol and distilled water. To the acidified aqueous alcohol was added 681 grams ethyl cellulose. The pH of the mixture was 2.3. The ethyl cellulose contained 45.9% ethoxyl groups, and when dissolved in 115 a solvent consisting of 80 parts toluene and 20 parts ethanol, to give a 5% solution, the solution possessed a viscosity of 65 cps. at 25 °C. The mixture of ethyl cellulose, swelling medium and acid was heated to 75 °C. 120 The mixture was maintained at 75° C. for 1.5 hours. Then the liquor was drained off. The volume was restored by the addition of 4000 ml. fresh 35% isopropanol. After 0.5 hour at room temperature the liquor was 125 drained off again. This procedure was repeated three more times. On the fifth wash only

3500 ml. of the liquor was drained off. volume was restored by the addition of 3500 ml. fresh 35% isopropanol. After 0.5 hour 3000 ml. of the liquid was drained off. The 5 volume was restored by the addition of 300 ml. 35% isopropanol. After 0.5 hour 2500 ml. of the liquor was drained off. volume was restored by the addition of 2500 ml. 35% isopropanol. After 0.5 hour the 10 liquor was drained off. This procedure was repeated two more times to wash the ethyl cellulose free of the salts and the excess acid. Then to the last wash was added 5 grams ditert-amylphenol dissolved in 25 ml. 35% iso-15 propanol. The liquor was drained off and the material was vacuum dried. The following data were observed in examining the treated and untreated ethyl cellulose as described in Example 1:—

> Ethyl cellulose product - 4.9 6.1 Untreated ethyl cellulose - 18 8.5

Example 5. To 5000 ml. of aqueous 35% by weight iso-25 propanol was added 12 ml. of 1:1 hydrochloric acid. The dilute isopropanol was prepared from distilled isopropanol and distilled water. To the acidified aqueous alcohol was added 681 grams of ethyl cellulose. The pH of the mixture was 2.5. The ethyl cellulose contained 46.5% ethoxyl groups and when dissolved in a solvent consisting of 80 parts toluene and 20 parts, ethanol, to give a 5% solution, the solution possessed a viscosity of 97 cps. at 25° C. 85 The mixture of ethyl cellulose, swelling medium and acid was heated to 75° C. Then 3.0 grams of sodium chlorite dissolved in 400 ml. of 35% isopropanol was added. The mixture was maintained at 75° C. for 1.5 hours. 40 Then the liquor was drained off. The volume was restored by the addition of 4000 ml. of fresh 35% isopropanol. After 0.5 hour at room temperature, the liquor was drained off again. The volume was restored with 3000 ml. of 45 fresh 35% isopropanol. The procedure was repeated five more times to wash the ethyl cellulose free of salts and the excess acid. Then to the last wash was added 5 grams of di-tert-amylphenol dissolved in 25 ml. of 35% iso-50 propanol. The liquid was drained off and the material was vacuum dried and tested as in the first example.

Color % Haze
Ethyl cellulose product - 3.5 4.0

55 Untreated ethyl cellulose - 18 9

Example 6.

The procedure described in Example 1 was employed except that the bleaching with sodium chlorite was effected at room tempera60 ture instead of at 75° C., and the pH of the aqueous alcohol was adjusted to 2.9 with hydrochloric acid.

| | Color | % Haze | |
|-----------------------------|-------|--------|----|
| Ethyl cellulose product - | 4.1 | 4.0 | |
| Untreated ethyl cellulose - | 18 | 9 | 65 |

EXAMPLE 7.

To a glass container was added 2400 ml. of distilled water containing 1.5 grams of sodium chlorite. Then, 680 grams of ethyl cellulose containing 45.8% ethoxyl groups and 70 having a viscosity of 79 cps. at 25° C. was added. Then, 2140 ml. of 85% isopropanol containing 5 ml. of 1:1 sulfuric acid was added. The mixture was heated to 75° C. and maintained at this temperature for one hour. The 75 pH of this mixture was 2.2. The liquor was drained off and replaced with fresh 35% isopropanol. This was repeated to provide four more washes. The alcohol added for the last wash contained 6.8 grams of di-tert-amyl-80 phenol. The ethyl cellulose was vacuum dried and then tested as in the first example.

Ethyl cellulose product - 3.7 4.8 Untreated ethyl cellulose - 17 11 85

EXAMPLE 8.

A mixture of 1.36 grams of sodium chlorite, 1500 ml. of distilled water and 100 ml. of 90% ethanol was placed in a glass container. To this mixture was added 680 grams of ethyl 90 cellulose containing 45.8% ethoxyl groups and having a viscosity of 79 cps. at 25°C. Then a solution consisting of 1400 ml. 90% ethanol and 6 ml. 1: 1 hydrochloric acid was added. This produced a slurry with a ph of 2.9. The 95 mass was heated to raise the temperature to 75°C., and this temperature was maintained for 0.5 hour. The liquor was drained off and 2600 ml. of fresh 50% ethanol was added. After one hour at room temperature the liquor 100 was drained off again and 2600 ml. of fresh 50% ethanol was added. Three more washes were applied in this manner, the alcohol for the final wash containing 6.8 grams of ditert-amylphenol. The ethyl cellulose was 105 vacuum dried and tested as in Example 1.

Ethyl cellulose product - 5.3 6.6 Untreated ethyl cellulose - 17 11

I. The method for producing a colorstable, viscosity-stable cellulose ether which comprises contacting an organic solvent-soluble cellulose ether (as hereinbefore defined) with an acidified aqueous swelling medium, separating said 115 cellulose ether from said swelling medium, stabilizing the cellulose ether by adding an unacidified aqueous swelling medium and a minor proportion of a phenol and separating said cellulose ether from said unacidified swelling medium, the phenol being absorbed in the cellulose ether.

2. The method claimed in Claim 1, wherein said cellulose ether is contacted with a water-

soluble inorganic chlorite bleaching agent in the presence of said acidified aqueous swelling medium.

 The method claimed in Claim 2, where in the chlorite is a metal chlorite, preferably an alkali metal chlorite, e.g. sodium chlorite.

4. The method claimed in Claim 1, wherein the swelling medium is an aqueous solution of a lower aliphatic alcohol, e.g. isopropanol, 10 containing from 10 to 50 weight per cent of said alcohol.

5. The method claimed in any of the preceding claims wherein the first swelling medium is acidified to a pH within the range of 1.5 to

15 3.5 with a strong mineral acid.

6. The method claimed in any of the preceding Claims, wherein the cellulose ether is ethyl cellulose.

7. The method claimed in any of the pre-20 ceding Claims, wherein the phenol is di-tert-

amylphenol.
8. The method claimed in any of Claims 3 to 7, wherein from 0.1 to 2 weight per cent of the metal chlorite based on the cellulose

25 ether is employed.

9. The method claimed in any of the preceding Claims, wherein said cellulose ether is contacted with said acidified aqueous swelling agent for a period of time up to 20 hours.

30 10. The method claimed in any of Claims 3 to 8, which comprises forming a mixture of said cellulose ether, acidified aqueous swelling medium and bleaching agent at a temperature within the range of 0° to 130° C. and below 35 the boiling point of said swelling medium.

11. The method claimed in Claim 10, wherein said mixture is maintained at said pH and temperature for a period of time within the range of 0.1 to 10 hours.

12. A color-stable, viscosity-stable cellulose 40 ether composition, said composition having been prepared by contacting an organic solvent-soluble cellulose ether with an acidified aqueous swelling medium for said cellulose ether, separating said cellulose ether from said 45 swelling medium, and stabilizing the cellulose ether by adding an unacidified aqueous swelling medium and a minor proportion of a phenol and separating said cellulose ether from said unacidified swelling medium, the phenol 50 being absorbed in the cellulose ether.

13. A cellulose ether as claimed in Claim 12, wherein said celluose other has been contacted by a water-soluble inorganic chlorite bleaching agent, preferably a metal chlorite. 55

14. A cellulose ether as claimed in Claim 12 or 13 wherein the cellulose ether is ethyl cellulose substantially free of alkaline constituents and having an ash content not in excess of 0.02 by weight.

15. A cellulose ether as claimed in Claim 12, wherein the swelling medium employed is a lower aliphatic alcohol such as isopropanol.

16. A cellulose ether as claimed in any of Claims 12 to 15, wherein from 0.5 to 1% by 65 weight of di-tert-amylphenol based on the cellulose ether is employed.

17. The method of producing a color-stable, viscosity-stable cellulose ether, substantially as hereinbefore described with reference to the 70

foregoing examples.

18. A color-stable viscosity-stable cellulose ether substantially as hereinbefore described.

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Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press.—1955 Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.